

# The Coking Properties of Coal at Elevated Pressures

Michael S. Lancet  
Frank A. Sim  
George P. Curran

Conoco Coal Development Company  
Research Division  
Library, PA 15129

## INTRODUCTION

Conoco's experience with the gasification of Pittsburgh No. 8 and Ohio No. 9 coals in Westfield, Scotland has suggested that the coking properties of these coals, and other Eastern U.S. coals by inference, under increased pressure are different from those normally measured at one atmosphere.

Convinced that the initial coal-based commercial synfuels plants in the U.S. will be gasification plants, Conoco has invested in the equipment necessary to carry out this study.

The work reported herein involves the operation of a pressurized coker, a pressurized Gieseler Plastometer and a device for measuring the Pressurized Swelling Index (PSI). The results of testing various coals by these methods are particularly useful in the evaluation of said coals for use in both dry and wet bottomed Lurgi gasifiers.

Seven coals, covering a fairly wide range of ranks and characteristics, were included in this work. These included three Eastern U.S. bituminous coals, EPRI-Champion and Westland from the Pittsburgh No. 8 seam and Noble County from the Ohio No. 9 seam. Also included were an Illinois No. 6 bituminous coal, Burning Star, a Western U.S. subbituminous coal, Upper Hiawatha, from Utah, and two bituminous coals, Frances and Rossington from the U.K. Four of these coals, EPRI-Champion, Noble County, Frances and Rossington were actually run in the BGC/Lurgi slagging gasifier during various programs in Westfield, Scotland.

## EXPERIMENTAL

Drum sized samples of each coal were prepared for testing by the flow sheet shown in Figure 1. All samples were stored in sealed plastic bags until ground finer than 1/4" after which time they were saved in a CO<sub>2</sub> atmosphere in a dry ice chest.

The parting material or gob from each coal sample was saved and sized as needed for each test where gob addition was called for. Several of the samples, as received, contained no gob, hence waste material from another mine in the same seam or, if that was not available, parting material from a similar coal sample was used. The following gob was used with each coal as listed below:

<u>Coal</u>	<u>Gob</u>
EPRI	Westland
Westland	Westland
Noble County	Egypt Valley - Ohio No. 9
Burning Star	Hillsboro - Illinois No. 6
Frances	None
Rossington	Hillsboro
Upper Hiawatha,	Upper Hiawatha

The proximate and ultimate analyses of these coals are given in Table 1. Also shown in this table are the heating values, ASTM Gieseler fluidity and Free Swelling Index (FSI), the Hardgrove grindability and a strength index for each coal. These represent the normally measured physical and chemical properties of coal.

The Gieseler fluidity in DDPM and the Free Swelling Index (FSI) reported in Table 1 are the standard ASTM test values for each coal.

The relative strength of each coal was determined by the CCDC mini drum method which consists of tumbling a 10 g sample in a specially designed 8" diameter tumbler with two 180° opposed internal vanes. A composite mini drum index was calculated for each coal which can be used to compare the relative strengths of coals and/or cokes. The higher the number the stronger the material. The same test was used to measure the strengths of all cokes produced in the pressure coker runs described later.

The coal strengths, ranging from a low of 0.81 for the Noble County coal to 0.94 for the Frances, are quite high. In almost every case the strength of each coal is greater than that of the cokes derived from them.

The grindability numbers are consistent with the strength indices with Frances at 38.6 being the hardest to grind and the strongest from a strength index standpoint. Based on the grindability all the coals are physically rugged and should pose no problems upon feeding to a Lurgi gasifier.

#### Pressurized Coal Fluidity Studies

The measurement of the fluidity of coals as they are heated through 350 to 500°C has been of significant benefit to the steel industry in predicting the performance of various coals in slot ovens. The standard method of determining coal fluidity is via the Gieseler plastometer. This test, normally carried out at 1 atm total pressure in air, consists of applying a constant torque to a rubble arm stirrer, packed in a sample of finely ground coal, and measuring the rate of rotation of the stirrer as the sample is heated at a constant rate (usually 3°C/min) through the plastic zone.

In general, coals of higher rank (except anthracite) are found to be more fluid by this test than those of lower rank. Little is known, however, about the fluidity of coals at elevated pressures and in gas atmospheres other than air.<sup>(1,2)</sup> This becomes of particular interest for moving bed coal gasifiers where raw coal is introduced at rather high temperatures and at elevated pressures of reducing gas. Hence, the work described herein was aimed at determining the effect of these variables, as well as higher heating rates, on the fluidities of different coals. Table 2 lists the parameters tested in this work.

#### Apparatus:

The basic equipment used in this work has been described previously.<sup>(2)</sup> Both the heating rate and the torque are continuously variable on this machine between the values of 0 and 6°C/minute and 0 and 720 g.cm, respectively. The variable heating rate permits examination of the effect of this parameter on coal fluidity whereas normal Gieseler operation is at a fixed heating rate of 3°C/minute. The capability of lowering the torque below the standard value of 100 g.cm permits measurement of fluidities far in excess of the normal 30,000 DDPM maximum.

#### Procedure:

Hand picked lumps of coal were ground in air to -35 mesh and either tested immediately or stored in a CO<sub>2</sub> atmosphere in a dry-ice chest. Coal samples of this kind are identified in this report as "clean coal". When specified by the program, "clean" coal samples were doped with gob ground to -35 mesh. Frances coal was not doped due to the clean nature of this seam. Tar was added to the coal and gob mixture as a solution in toluene. The coal-gob-tar slurry was then placed in a vacuum oven and the toluene was evaporated at about 50°C.

A modified ASTM D-2639-71 "Standard Method of Test for Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer" was employed. As opposed to the ASTM standard method, the majority of the runs in this study were done at a heating rate of 6°C/minute and only one for each coal was made at the standard rate of 3°C/minute. Also, the standard torque of 1.40 oz-in (100 gm-cm) was used only when the maximum fluidity of the coal in question was within the range of the apparatus, i.e., 0-28,000 DDPM. In the cases where the coal fluidity exceeded the operating range of the instrument, "low" torques, i.e., 0.45 oz-inch (32.4 g-cm) or 0.26 oz-inch (18.7 g-cm), were employed and the results were corrected by factors of 3.1 or 5.4, which are the ratios of the standard torque and the lower torques, (1.4/0.45 and 1.4/0.26), respectively. A considerable degree of uncertainty may be associated with the numerical value of the high torque/low torque correction factor. The actual ratio of the torques is theoretically correct for Newtonian fluids. However, since each coal in its plastic state may deviate from Newtonian behavior to a different degree, the values of 3.1 or 5.4 may only be considered to be approximations to the true values.

When the test was to be performed in a gas mixture containing 18 or 31.5 vol % H<sub>2</sub> at a total pressure of 350 psig, the pressure vessel containing the plastometer was evacuated and then pressurized with hydrogen to 51 psig and 100 psig, respectively. The hydrogen was then diluted to the desired degree by pressurizing the vessel up to 350 psig with prepurified nitrogen. The remainder of the runs were carried out in a prepurified nitrogen atmosphere or in air.

#### Results and Discussion:

Tables 3 through 6 show the effects of the studied variables, i.e., of the heating rate, gas composition, gob content, tar content, and nitrogen pressure, on the fluidity of the coals studied. Figures 2 through 6 are graphical representations of the data obtained on the fluidities of the seven coals in this program.

The following conclusions may be drawn from these data:

1. In all cases, an increase in the heating rate resulted in substantially increased fluidity (Figures 2 and 3). This effect is directly comparable to and in complete agreement with that observed by Van Krevelen, et.al.<sup>(3)</sup>
2. In all cases, except that of Frances and Upper Hiawatha coals, the fluidity of the coal is moderately sensitive to hydrogen partial

pressure at 350 psig total pressure (Figure 4). The effects of all the variables - except the heating rate - on the fluidity of Frances and Upper Hiawatha coal is so small that these coals may be considered non-fluid.

3. The observed effect of an increase in fluidity with an increase in total, or nitrogen pressure, Table 6, is consistent with work done previously on other coals<sup>(1,2)</sup> in this pressure range.
4. Increasing the gob content in all coals reduced the fluidity in all cases. Frances coal was not subject to this type of test.
5. The addition of 4 wt % of Pittsburgh No. 8 derived tar significantly increased the fluidity of all coals except the Frances and Upper Hiawatha.

The results of this work on the effect of both total pressure and hydrogen partial pressure, qualitatively support the conclusion of Lewellen,<sup>(4)</sup> however, the available data are insufficient to quantitatively test this model. To fully test this model would require a much wider range of both pressures ( $10^{-2}$ - $10^3$  atm) and heating rates (up to  $10^4$ °C/min) than is currently possible.

The qualitative correlations of the fluidity with the experimental parameters, as discussed above, suggest that with additional data, especially from work on other coals, a meaningful predictive correlation of these data may eventually be possible. Initial attempts to correlate the observed fluidity data with various physical and chemical properties have been encouraging. There is strong evidence which suggests that with data from several additional coals a correlation of fluidity with various petrographic features may be obtained.

#### Pressurized Swelling Index

The free swelling index, FSI, of coal as defined by the ASTM<sup>(5)</sup> is another valuable tool of the steel industry. This test consists of rapidly heating a finely ground coal sample to about 800°C and observing the degree to which the coal cokes and swells. This test, as in the case of standard Gieseler work, is carried out at one atmosphere in air. The values for the ASTM FSI's for each coal are given in Table 1. For the present work it was believed that a similar test under simulated gasifier conditions might be of value in predicting the behavior of coals as fed to a moving bed gasifier.

While the pressurized swelling tests carried out in this work are as close to the ASTM standard method as possible, one major difference was unavoidable. Due to the use of elevated pressures the heating rate of this test is not the same as that prescribed by the ASTM standard method.<sup>(5)</sup> Furthermore, the heating rate at each test condition was slightly different due to the difference in pressure and thermal conductivity of the gases used. The heating rate was, however, the same for each coal at the same test condition and conclusions drawn from such comparisons should be valid. In general the coke buttons produced in the CCDC pressurized swelling index (PSI) test are significantly less voluminous than those of the ASTM test.

#### Apparatus:

The equipment for this test, as shown schematically in Figure 7, consists of an electrically heated steel core enclosed in a pressure shell. A sample crucible holder assembly is attached to a 1/4" lifting rod which passes through a packing gland at the top of the vessel. Strategically located thermocouples permit continuous monitoring of the core and crucible temperature.

#### Procedure:

A 1.00 gm sample of coal, ground to -60 mesh, was placed in the test crucible, covered with a lid and positioned in the holder. The vessel was then closed, sealed and evacuated in order to displace air and then flooded or pressurized with the desired gas. After the required pressure was attained, the lifting rod with the holder and the crucible was pushed down onto the core surface which had previously been equilibrated at  $1500^{\circ} \pm 5^{\circ}\text{F}$ . Temperatures of the core were recorded every 20 seconds beginning with the time when the crucible reached the core. The crucible was lifted after 5 minutes, the apparatus was depressurized, purged with nitrogen and the crucible was removed. The coke button was carefully taken from the crucible and weighed. The pores in the button were plugged by dipping in molten paraffin and the volume was measured by water displacement. The swelling index was defined as the button volume in milliliters. For comparison purposes, the ASTM FSI profile number is very nearly the button volume in ml. Each experiment was repeated 4 to 5 times due to the variability in the button volume, and the value reported for each is the average of all runs. Normally the standard deviation of this average was less than  $\pm 10\%$ .

Temperature profiles were measured in order to ascertain that the samples were subject to comparable heating rates. The initial heating rates are quite high ( $1000\text{-}1200^{\circ}\text{F}/\text{min}$ ) and the temperature stabilizes after 3 minutes. The heating rate in both hydrogen and nitrogen is nearly the same.

#### Experimental Results:

Each of the seven coals in the program was tested by this method. Tar obtained during operation of the Westfield gasifier with Pittsburgh No. 8 coal was used for the tar addition experiments with all tested coals.

The coals and their mixtures with gob and tar were tested at the conditions shown in Table 7.

Table 8 shows the effects of the sample composition on the swelling of different coals at 365 psia total and 115 psia hydrogen partial pressure. Figure 8 is a graphical representation of these results.

The experimental results lead to the following conclusions:

1. There is little difference between the swelling properties of the Noble County, Ohio No. 9 coal and the two Pittsburgh seam coals, EPRI-Champion and Westland, at the test conditions, i.e., at  $1500^{\circ} \pm 5^{\circ}\text{F}$ , 350 psig total pressure and 115 psia hydrogen partial pressure. This is in marked contrast to the 1 atm ASTM FSI data (Table 1) where the two Pittsburgh seam coals have FSI values of 7

while the Ohio No. 9 has an FSI of only 4! The Burning Star-Illinois No. 6 coke buttons and those made of Rossington coal were considerably less voluminous than those of the EPRI coal. Frances and Upper Hiawatha coals are virtually non swelling.

With the exception of the Noble County coal, the order of the pressurized swelling index values is the same as that of the ASTM FSI values. The apparent greater effect of gasifier conditions on the Ohio No. 9 coal than on the Pittsburgh No. 8 coals may help explain some of the unexpected operating difficulties which occurred when the Noble County coal was fed to the BGC/Lurgi slagging gasifier in the DOE sponsored Westfield trials. The observed differences in the caking and swelling of the Ohio No. 9 coal at Westfield versus those predicted by standard ASTM tests was one key factor which eventually led to the present CCDC feedstock evaluation program.

It is interesting that increased hydrogen pressure and total pressure has a larger relative effect on the swelling of the Noble County coal than on its fluidity. The increase in the fluidity of this coal is increased by gasifier conditions by about the same factor as are the two Pittsburgh seam coals.

2. Doping with gob significantly reduces the swelling of all the coals tested except the inactive Upper Hiawatha coal where the gob addition had no effect. This observed effect is essentially that expected from the diluent effect of the inert gob.
3. The addition of tar to the bituminous coals when they have been doped with 10 wt % gob, considerably increases the volume of coke buttons made of these coals (Table 9). Tar addition to the Utah subbituminous coal does not have any effect, while the same tar addition to Frances coal increases its very low swelling activity by about 10%.

The addition of even 4% tar has little effect on the Noble County coal (Table 9). This, coupled with the much larger observed effect of operating at gasifier conditions discussed above, suggests that the coal-derived tar content of this coal may be significantly higher than that of the other coals. This, however, is not confirmed by the coker yield data to be discussed later. It is possible that such differences in the yield structure would be masked by reactions between the coal-derived tar and added hydrogen or even pyrolysis-derived gases. More coal-derived tar from the Noble County coal could also explain the observation that this coal has the highest ASTM Gieseler fluidity of any coal tested (Table 1).

4. The effect of hydrogen partial pressure on the swelling of all the coals tested, doped with 10% gob, is negligible. There was little difference between the buttons produced in 100% N<sub>2</sub> at 350 psig and those made in 31.5% H<sub>2</sub>-68.5% N<sub>2</sub> at 350 psig total pressure. The use of a gas containing 18 vol % H<sub>2</sub> also did not affect the volumes of the coke buttons of EPRI coal containing 10% gob. The effect of

the total pressure on the swelling properties was studied only with EPRI coal doped with 10% gob. Prepurified nitrogen was used and the results suggest there is little or no effect of the total pressure on the swelling properties of this coal.

While the effect of both hydrogen pressure and total pressure on the button volume is small they have a marked effect on the shape of the coke buttons. Coking a highly caking Eastern U.S. coal in the pressurized FSI apparatus produces, at low pressure, a button with a smooth rounded top surface. At pressures above about 150 psig, however, one or more appendages which resemble stalagmites begin to appear on the top surface of the button.

This effect is not observed when lower rank coals are processed in the device. Figure 9 shows a comparison of the effect of pressure on the shape of the buttons produced from a typical Pittsburgh No. 8 seam coal with those produced from a less active, Illinois Basin, coal.

Apparently the pressure tends to prevent the whole top surface from rising as seems to be the case at atmospheric pressure, but internal pressures are built up which are eventually released through the observed stalagmite growth.

As discussed earlier, there is encouraging evidence that more data on these properties, to be obtained by running more coals through the program, will lead to a predictive correlation of the swelling properties based upon petrographic parameters.

#### Pressurized Coker Studies

The CCDC pressurized coker is a useful tool for testing several parameters which are associated with coke formation in the upper part of a Lurgi gasifier. Especially important in this respect is the friability which may be a key to successful stirrer design in future plants. This property is studied via the mini drum tumbler test described earlier. Other coking properties which are studied are the effect of washing, i.e., the effect of non-coal impurities, on the coke formed under pressure and the effect of recycle tar addition on coke formation. An understanding of these effects will be important for gasifier and coal preparation facilities design requirements.

All coals included in this program were subjected to testing in the pressurized coker system to ascertain the effects of the total operating pressure, the hydrogen partial pressure, gob addition and tar addition on the coke strength and coke density. Product yields were determined for the runs made with clean coal. Also, chemical analysis of gases, tars and cokes were obtained for the clean coal runs and the effects of the above mentioned independent variables on the methane, carbon monoxide and hydrogen contents of the product gas was investigated.

#### Apparatus:

The pressurized coker is a 48" long, 2" diameter, double x wall pipe made of Alonized 316 SS. The coker tube is capable of being rotated about an axis located at the tube center as shown in Figure 10. The outlet half of the

coker tube is electrically heated with 6, 520 watt, resistance heaters. Skin and internal temperatures are continuously monitored and recorded on a strip chart. The tar trap is a small water-cooled pressure vessel. A dip tube connected to the detachable lid reaches to the bottom of a teflon bottle tightly fitted into the tar trap body. Glass wool, placed in the annular space between the dip tube and the bottle neck acts as a demister. An opening is provided in the tar trap lid for gas withdrawal.

A pressure control valve is located downstream of the tar trap and is operated by a recording pressure controller and a pressure transmitter which is connected to the gas inlet piping of the coker.

A dry ice temperature cold trap is located downstream of the pressure control valve. Gas sampling bags and gas meters are provided on the tail end of the system.

#### Procedure:

A 100 gram sample of sized coal or of a coal and gob mixture is inserted into the cool inlet side of the coker while the body is kept in the horizontal position. When tar was to be added to the sample a solution of the desired weight of tar in 5 ml of toluene was prepared. The solids were immersed in this solution, the toluene was evaporated in a vacuum oven at 50°C and the sample was chilled in an ice chest prior to loading into the coker.

After the sample was charged into the coker, the inlet flange was attached and the whole system purged with nitrogen at atmospheric pressure. Heat-up was begun and when the coker thermowell temperature approached 1300°F, the nitrogen purge was switched to the desired gas mixture and the gas flow rate and operating pressures were set as specified for the desired run.

When the coker bed temperature reached 1472°F (800°C) an inlet gas sample was taken by opening and closing previously evacuated gas sample bomb. A split stream of the off gas was diverted into a gas sample bag and the coker body was tilted into the vertical position. The sample dropped into the hot zone of the coker and the coking process was begun. This shock heating under pressure attempts to simulate coal falling into the top of a moving bed gasifier.

The power was turned off after 15 minutes running time and the off gas sample was collected for a total of one hour. The system was then depressurized, the cold trap closed, and the coker was vented and kept under a small nitrogen purge until the apparatus cooled down. The tar trap was then disconnected and the tar removed, separated from the water phase and weighed. The coke was removed and weighed after the coker internal temperature had dropped below 200°F.

#### Results and Discussion:

A total of ten different tests were devised to investigate the effects of the total operating pressure, the hydrogen partial pressure and of the sample composition on coke strength, coke density, coking yields and on the composition of the products. Table 9 is a list of test conditions. All the tests were made on coal sized 3/4" x 1/4" except test 10. Experiments were performed in atmospheres of either 100% prepurified nitrogen or mixtures of prepurified nitrogen with hydrogen. It should be mentioned that the tests identified as No. 2 and No. 7 were done for EPRI-Champion coal only.



A comparison of the results obtained from tests Nos. 3, 4 and 5 shows the effect of total pressure, namely, atmospheric, 215 psig and 365 psig on coke strength and mercury density of the coke. Similarly, a comparison of tests Nos. 2, 3 and 6 shows the effects of 0, 65 and 115 psia hydrogen partial pressure (the balance being nitrogen) on the same variables. Tests Nos. 1, 6 and 9 compare the effects of gob addition to the coal, with test No. 1 investigating "clean" coal, No. 6, coal doped with 10% gob and No. 9, coal doped with 20% gob. Tests Nos. 7 and 8 study the effect on the above mentioned properties of adding 5 and 10 wt % tar to a coal sample doped with 10% gob. Test No. 10 was run on a "clean coal" sample sized 1/4" x 12 mesh at 365 psia total pressure and 115 psia of hydrogen partial pressure. The finer size of the feed coal for this test allowed for a more homogeneous sample than was possible when 3/4" x 1/4" coal was used which, in turn, permitted better data on yield and coke composition to be obtained.

The inherent variability of the coal and particularly that of the gob samples used rendered any yield calculations based on these feed mixtures useless. Hence, only those runs made with undoped clean coal were used for yield determinations.

Figure 11 is a plot of the effect of various parameters on the coke strength. The plotted data are averages of all runs made at each set of conditions. The following conclusions may be drawn from this figure:

1. The strength of the coke made from the low fluidity coals (Frances and Upper Hiawatha) and from the medium fluidity coals (Rossington and Burning Star) is inversely proportional to total pressure.

The effect of increasing pressure on the strength of coke made from the high fluidity coals (EPRI, Westland and Noble County) is negligible.

2. The effect of hydrogen partial pressure on the coke strength is small and variable (Figure 11).
3. The results related to the effect on the coke strength of doping clean coal with 10% and 20% gob are inconclusive. The coke strength decreases or remains basically unchanged in six of seven cases, the exception being the Burning Star coal.
4. The addition of 10 wt % tar to coal previously doped with 10 wt % gob caused a slight increase in the strength of the coke produced in most cases with the exception of the Upper Hiawatha and Burning Star coals.

Figure 12 is a plot of the effects of pressure, gas composition and gob and tar addition on the coke density. These are particle densities as determined by mercury displacement at one atmosphere. The data shown in this figure suggest that:

1. The coke density remains the same or decreases very slightly when the total pressure is increased from atmospheric to 350 psig.

2. The density of the coke produced from the studied coals depends little on the hydrogen partial pressure. However, a slight increase in density with increased hydrogen pressure is seen for the high fluidity coals.
3. As expected increased gob contents lead to increased particle densities.
4. The addition of 10 wt % tar to low fluidity coals did not have any effect on the density of the coke. However, the same addition to medium fluidity coals doped with 10% gob caused the coke density to increase significantly. A slight increase is observed for two of the three high fluidity coals (EPRI and Westland) while a decrease was found for the Noble County coal.

Interestingly, this rather complex picture of coke strength and density may, as appears to be the case for the fluidity and swelling data, be explainable in terms of a few petrographic parameters. More data on different coals will show just how good a correlation can be obtained.

Yield data were collected for each of the over 100 coker runs in this program, however, as mentioned earlier the majority of these data are of little value. Only the data obtained with clean coal samples give useful yield data. A summary of the yield data from the runs made at simulated gasifier conditions (i.e., 365 psia total pressure and 115 psia hydrogen pressure) on 1/4" x 12 mesh coal is given in Table 10. These data are based on the average measured coke, tar and liquid yields for all runs at these conditions for each coal (on a moisture and ash free basis), with the gas yield calculated by difference.

In all cases the coke yield at simulated gasifier conditions of 365 psia total pressure and 115 psia hydrogen partial pressure is greater than the fixed carbon content determined for each coal at 1 atm (Table 1). This is not surprising since at elevated pressure the release of volatile components is retarded.<sup>(1,2,3)</sup> The slowing of volatile release allows more of this material to be converted to coke at the expense of liquid and/or gas yield.

The yield data for Frances coal, Table 11, where only clean coal was used, shows that both coke and gas yields are increased at the expense of the liquid yield in going from 1 atm N<sub>2</sub> pressure to 365 psia of N<sub>2</sub>. Here the coke yield increases from ~ 65% to ~ 67.5% and the gas yield increases from ~ 25% to 29% while the liquid yield decreases from ~ 10% to ~ 4%. Possibly liquid evolution repression can account for both the increased coke and gas yields. If the liquid is held in the coal matrix some of it is coked and some is further converted to gas by the longer residence time at elevated temperatures thus reducing the overall liquid yield and increasing the coke and gas yields. At elevated hydrogen pressures (115 psia and 365 psia total pressure) the coke yield is about the same as when 365 psia of nitrogen is used, however, the liquid yield is greatly enhanced at the expense of the gas yield. Undoubtedly reaction between the added hydrogen and the coal are responsible for the higher liquid yield.

Figure 13 shows a comparison of produced gas composition with respect to CH<sub>4</sub>, CO and H<sub>2</sub> at various conditions. Since most of these data were obtained with coal doped with gob the results may not be as reliable as if only clean coal

was used, however, general trends are not expected to differ from those observed here. The methane content of the product gas is 50-100% higher than for the case with no added hydrogen while the hydrogen content of the product is considerably lower.

Qualitatively this suggests that hydrogen evolved from the coal escapes before it has time to react further with the remaining coal matrix. The CO content of the product appears virtually unaffected by pressure and the addition of gob and/or tar has little or no effect on the gas composition.

#### CONCLUSIONS

The experimental data presented herein should provide a start on the required data base which will ultimately permit the prediction of the coking properties of coals being considered as potential gasifier feedstocks. The limited data obtained pertaining to pyrolysis yields and gas composition are encouraging and suggest that accurate gas yield predictions may also be possible at the conclusion of this program. The effects of process variables on coal swelling and fluidity has been fairly well established, but more work is needed to get predictive mathematical correlations of these as well as coking and pyrolysis properties of coals.

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#### REFERENCES

1. Kaiho, M. and Toda, Y., Changes in Thermoplastic Properties of Coal Under Pressure of Various Gases. Fuel 58, 397 (May, 1979).
2. Lancet, M. S. and Sim, F. A., The Effect of Pressure and Gas Composition on the Fluidity of Pittsburgh No. 8 Coal, Preprints of Fuel Chemistry Div. ACS, Vol 26, No. 3 (August, 1981).
3. Van Krevelen, D. W., Huntjens, F. S. and Dormans, H. N. M., Chemical Structures and Properties of Coal XVI - - Plastic Behavior on Heating, Fuel 35, 462 (1955).
4. Lewellen, P. C., Product Decomposition Effects in Coal Pyrolysis, Masters Thesis, Department of Chemical Engineering, MIT, (1975).
5. ASTM Standard Method D720-67, Free Swelling Index of Coal (1977).

Table 1  
Composition of Feed Coals

Coal	<i>EPR Champion</i>	<i>Westland</i>	<i>Noble County</i>	<i>Burning Star</i>	<i>Frances</i>	<i>Rossington</i>	<i>Upper Hiawatha</i>
Origin	Pgh. No. 8 Seam	Pgh. No. 8 Seam	Ohio No. 9 Seam	Ill. No. 6 Seam	Scotland	England	Utah
<u>Moisture</u> % As Analyzed	2.28	2.02	2.68	9.99	6.62	5.81	7.39
<u>Proximate, Dry</u>							
Volatile Matter, %	37.93	36.28	44.36	42.94	38.84	38.01	41.74
Fixed Carbon	49.94	52.21	46.10	43.52	56.93	59.04	50.36
Ash	12.13	11.51	9.54	13.54	4.23	2.95	7.90
<u>Ultimate, Dry</u>							
Hydrogen, %	4.93	4.84	5.16	4.65	5.07	5.06	4.94
Carbon	72.52	73.13	72.70	68.00	78.73	79.13	72.08
Nitrogen	1.45	1.51	1.05	1.31	1.60	1.66	1.25
Oxygen (Diff.)	6.80	7.16	8.12	9.26	9.93	9.68	13.35
Sulfur	2.17	1.85	3.43	3.24	0.44	1.52	0.48
Ash	12.13	11.51	9.54	13.54	4.23	2.95	7.90
<u>HHV, Dry</u> Btu/lb	12,955	12,990	13,370	12,165	13,790	14,195	12,688
<u>Ultimate, MAF</u>							
Hydrogen, %	5.61	5.47	5.70	5.38	5.29	5.21	5.36
Carbon	82.53	82.64	80.37	78.65	82.21	81.54	78.26
Nitrogen	1.65	1.71	1.16	1.52	1.67	1.71	1.36
Oxygen (Diff.)	7.74	8.09	8.98	10.71	10.37	9.97	14.50
Sulfur	2.47	2.09	3.79	3.75	0.46	1.57	0.52
Gieseler Fluidity, (DDPM)	5,300	15,800	27,000	4.7	1.0	5.3	0
Free Swelling Index (FSI)	7	7	4	3.5	1.5	3.5	1
Hardgrove Grindability	52.9	62.9	49.7	48.7	38.6	48.3	45.6
Strength Index	0.849	0.886	0.806	0.831	0.942	0.847	0.884

Table 2  
List of Testing Conditions  
for Pressurized Coal Fluidity Studies

Test No.	Sample Composition	Testing Pressure, psig	Gas Composition	Heating Rate, °C./Min
0	Clean Coal	0	Air	6
1	Clean Coal	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6
2	Clean Coal	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6
3	90 Wt % Coal + 10 Wt % Gob	350	100% N <sub>2</sub>	6
4(1)	90 Wt % Coal + 10 Wt % Gob	350	18% H <sub>2</sub> , Bal. N <sub>2</sub>	6
5	90 Wt % Coal + 10 Wt % Gob	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6
6(1)	80 Wt % Coal + 10 Wt % Gob + 1 Wt % Tar	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6
7	80 Wt % Coal + 10 Wt % Gob + 4 Wt % Tar	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6
8(1)	90 Wt % Coal + 10 Wt % Gob	200	100% N <sub>2</sub>	6
9	80 Wt % Coal + 20 Wt % Gob	350	31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	6

(1) Test performed on EPRJ-Champion, Pittsburgh No. 8 Seam coal only.

Table 3  
The Effect of the Heating Rate  
on the Fluidity of Various Coals

	EPRI-Champion Pittsburgh No. 8 Seam	Westland Pittsburgh No. 8 Seam	Noble County Ohio No. 9 Seam	Burning Star Illinois No. 6 Seam	Frances Scotland	Hossington England	Upper Wisconsin Utah
	DDPM	DDPM	DDPM	DDPM	DDPM	DDPM	DDPM
Heating Rate 3°C./Min	25,500	6,300	50,200 <sup>(1)</sup>	50	3	520	2
6°C./Min	61,700 <sup>(1)</sup>	19,500	155,000 <sup>(1)</sup>	230	7	2,600	5

Test Conditions: Total Pressure = 365 Psia  
Hydrogen Partial Pressure = 115 Psia  
Sample Composition = Clean Coal

Reported values are averages of two duplicate runs.

(1) Torque correction factors used for fluidities higher than 30,000 DDPM at 100 g cm torque.

Table 4

The Effect of the Gas Composition  
on the Fluidity of Various Coals

Gas Composition	EPRI-Champion Pittsburgh No. 8 Seam DDPM	Westland Pittsburgh No. 8 Seam DDPM	Noble County Ohio No. 9 Seam DDPM	Burning Star Illinois No. 6 Seam DDPM	Frances Scotland DDPM	Rossington England DDPM	Upper Hiawatha Utah DDPM
100% Nitrogen	36,900 <sup>(1)</sup>	12,600	73,700 <sup>(1)</sup>	30	6	1,400	3
18% H <sub>2</sub> , Bal. N <sub>2</sub>	48,300 <sup>(1)</sup>	--	--	--	--	--	--
31.5% H <sub>2</sub> , Bal. N <sub>2</sub>	53,600 <sup>(1)</sup>	14,500	81,200 <sup>(1)</sup>	80	7	1,800	3

Test Conditions: Total Pressure = 365 Psia  
Heating Rate = 6°C/min  
Sample Composition = 90 wt % coal + 10 wt % gob (except Frances coal)

Reported values are averages of two duplicate runs.

(1) Torque conversion factors used for fluidities higher than 30,000 DDPM at 100 g cm torque.

Table 5

The Effect of Gob and Tar Addition  
on the Fluidity of Various Coals

Sample Composition	EPRI-Champion Pittsburgh No. 8 Seam DDPM	Westland Pittsburgh No. 8 Seam DDPM	Noble County Ohio No. 9 Seam DDPM	Burning Star Illinois No. 6 Seam DDPM	Frances Scotland <sup>(1)</sup> DDPM	Rossington England DDPM	Upper Hiawatha Utah DDPM
Clean Coal	61,700 <sup>(2)</sup>	19,800	155,000 <sup>(2)</sup>	230	7	2,600	5
90 wt % Coal + 10 wt % Gob	53,600 <sup>(2)</sup>	14,500	81,200 <sup>(2)</sup>	80	--	1,800	3
80 wt % Coal + 20 wt % Gob	41,200 <sup>(2)</sup>	11,500	56,900 <sup>(2)</sup>	30	--	1,400	3
89 wt % Coal + 10 wt % Gob + 1 wt % Tar	57,300 <sup>(2)</sup>	--	--	--	--	--	--
86 wt % Coal + 10 wt % Gob + 4 wt % Tar	96,200 <sup>(2)</sup>	34,500 <sup>(2)</sup>	+161,500 <sup>(2)</sup>	540	11	3,500	4

Test Conditions: Total Pressure = 365 Psia  
Hydrogen Partial Pressure = 115 Psia  
Heating Rate = 6°C/min

Reported values are averages of two duplicate runs.

(1) No gob added to Frances coal.

(2) Torque conversion factors used for fluidities higher than 30,000 DDPM at 100 g cm torque.

Table 6

The Effect of the Nitrogen Pressure on the  
Fluidity of EPMI-Pittsburgh No. 8 Seam Coal

<u>Pressure, psig</u>	<u>Fluidity, DDPM</u>
200	27,600
350	36,900

Test Conditions: Sample Type = "clean" coal  
Torque = 32.4 g cm  
Heating Rate = 6°C/min

Remarks: Reported values are averages of duplicate runs. Torque  
conversion factor equal to 3.11 used to obtain fluidity  
values as shown.

Table 7

List of Testing Conditions for  
the Pressurized Swelling Index Determination

<u>Test</u>	<u>Sample</u>	<u>Pressure P<sub>total</sub> P<sub>sw</sub></u>	<u>P<sub>H<sub>2</sub></sub> P<sub>sw</sub></u>
1	Clean Coal	365	115
2	90 Wt % Coal + 10 Wt % Gob	365	0
3(1)	90 Wt % Coal + 10 Wt % Gob	365	65
4	90 Wt % Coal + 10 Wt % Gob	365	115
5(1)	89 Wt % Coal + 10 Wt % Gob + 1 Wt % Tar	365	115
6	86 Wt % Coal + 10 Wt % Gob + 1 Wt % Tar	365	115
7(1)	90 Wt % Coal + 10 Wt % Gob	215	0
8	80 Wt % Coal + 20 Wt % Gob	365	115

(1) These were run only for one coal (EPMI-Champion, Pittsburgh No. 8  
Seam coal).

Table 8  
Effect of Feed Composition  
on the Swelling of Different Coals

Sample Composition	Coal						
	EPRI Champion Pittsburgh No. 8	Westland Pittsburgh No. 8	Noble County Ohio No. 9	Burning Star Illinois No. 6	Frances Scotland	Rossington England	Upper Hiawatha Utah
Clean Coal	3.0	2.8	3.0	2.0	1.0	1.8	0.9
90 Wt % Coal + 10 Wt % Gob	2.4	1.9	2.3	1.8	--	1.6	0.9
80 Wt % Coal + 20 Wt % Gob	2.2	2.1	2.4	1.6	--	1.7	0.9
89 Wt % Coal + 10 Wt % Gob + 1 Wt % Tar	2.6	--	--	--	--	--	--
86 Wt % Coal + 10 Wt % Gob + 4 Wt % Tar	2.9	3.8	2.3	1.9	1.1	2.0	0.9

Swelling index is expressed as the average volume in ml of all coke buttons produced at given test conditions.

$P_{tot}$ : 365 psia  
 $P_{H_2}$ : 115 psia  
 $T$ :  $1500 \pm 5^\circ F$

Table 9  
List of Pressurized Coker Testing Conditions

Test No.	Total Testing Pressure, psia	Hydrogen Partial Pressure, psia	Sample Composition
1	365	115	Clean Coal
2(1)	365	65	90 Wt % Coal + 10 Wt % Gob
3	365	0	90 Wt % Coal + 10 Wt % Gob
4(1)	215	0	90 Wt % Coal + 10 Wt % Gob
5	15	0	90 Wt % Coal + 10 Wt % Gob
6	365	115	90 Wt % Coal + 10 Wt % Gob
7(1)	365	115	85 Wt % Coal + 10 Wt % Gob + 5 Wt % Tar
8	365	115	80 Wt % Coal + 10 Wt % Gob + 10 Wt % Tar
9	365	115	80 Wt % Coal + 20 Wt % Gob
10(2)	365	115	Clean Coal

- (1) Tests performed on EPRI-Champion, Pittsburgh No. 8 Seam coal only.  
 (2) Test No. 10 performed on 1/4" x 12 mesh sized coals, all other tests on  
 3/4" x 1/4" sized materials.



Table 10  
Coker Yield Data

<u>Feed Coal</u>	<u>Coke Yield</u> <u>(MAF Wt %)</u>	<u>Liquid Yield</u> <u>(MAF Wt %)</u>	<u>Gas Yield*</u> <u>(MAF Wt %)</u>
EPRI-Champion	64.9	14.0	21.1
Westland	65.5	12.4	22.0
Noble County	60.1	13.3	26.6
Burning Star	62.9	11.8	25.3
Frances	65.7	11.2	23.1
Rossington	62.1	15.4	22.5
Upper Hiawatha	57.8	12.3	29.9

T : 800°C  
P<sub>tot</sub>: 365 psia  
P<sub>H<sub>2</sub></sub> : 115 psia  
1/4" x 12 mesh clean coal

\* Gas yield is difference of coke and liquid from 100%.

Table 11  
Effect of Pressure and Gas Composition  
on the Coking Yields from Frances Coal

<u>Hydrogen</u> <u>Partial</u> <u>Pressure</u> <u>(psia)</u>	<u>Total</u> <u>Pressure</u> <u>(psia)</u>	<u>Tar</u> <u>Content in</u> <u>Feed (wt %)</u>	<u>Coke Yield</u> <u>(MAF Basis)</u> <u>(wt %)</u>	<u>Liquid Yield</u> <u>(MAF Basis)</u> <u>(wt %)</u>	<u>Gas Yield</u> <u>(MAF Basis)</u> <u>(wt %)</u>
115	365	0	65.6	10.0	24.4
115	365	0	65.7	12.3	22.0
115	365	0	67.0	--	--
115	365	0	67.8	9.5	22.7
115	365	0	67.8	11.3	20.6
0	365	0	67.5	3.7	28.8
0	15	0	65.0	9.8	25.2
0	15	0	65.0	--	--
0	15	0	65.4	--	--

Figure 1  
Sample Preparation Scheme

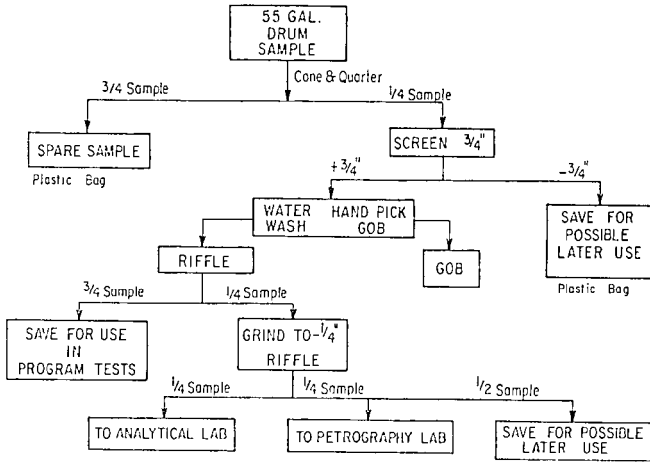


Figure 2  
EFFECT OF HEATING RATE ON THE FLUIDITY OF  
PITTSBURGH NO.8 AND OHIO NO.9 SEAM COALS

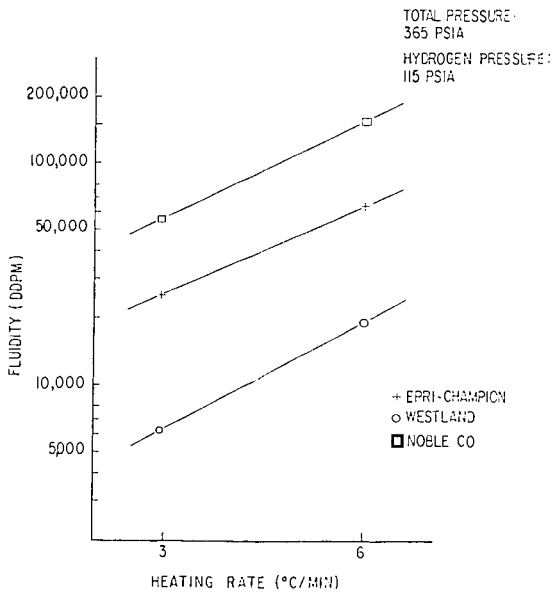


Figure 3  
EFFECT OF HEATING RATE ON THE FLUIDITY OF  
BURNING STAR AND ROSSINGTON COALS

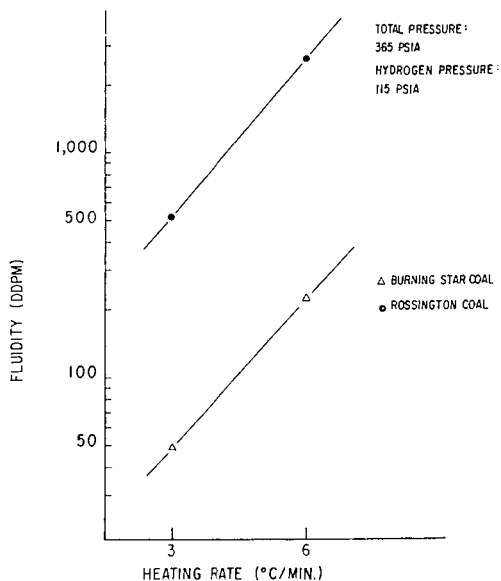


Figure 4  
EFFECT OF GAS COMPOSITION ON THE FLUIDITY OF  
VARIOUS COALS

TOTAL PRESSURE : 350 PSIG  
 HEATING RATE : 6°C MIN  
 SAMPLE : 90 WT % - 35M COAL + 10 WT % - 100M GOB

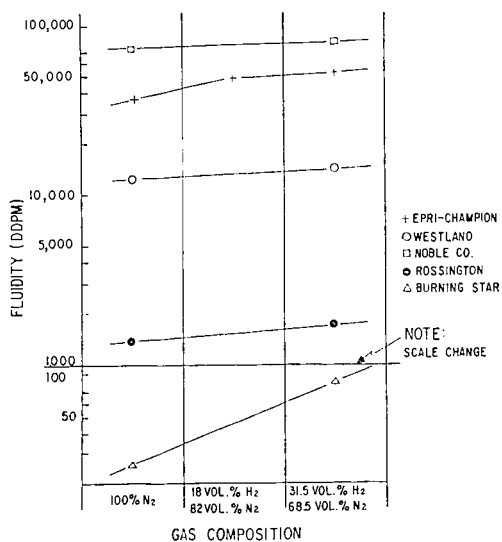


Figure 5  
EFFECT OF GOB ADDITION ON THE FLUIDITY OF  
VARIOUS COALS

TOTAL PRESSURE: 365 PSIA  
HYDROGEN PRESSURE: 115 PSIA  
HEATING RATE: 6°C/MIN

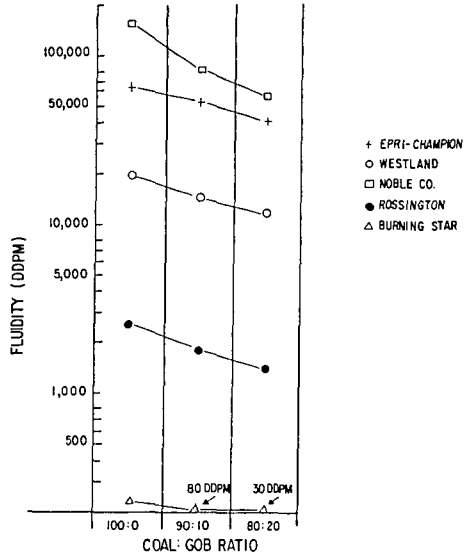


Figure 6  
EFFECT OF TAR ADDITION ON THE FLUIDITY OF  
VARIOUS COALS DOPED WITH 10% GOB.

TOTAL PRESSURE: 365 PSIA  
HYDROGEN PRESSURE: 115 PSIA  
HEATING RATE: 6°C/MIN

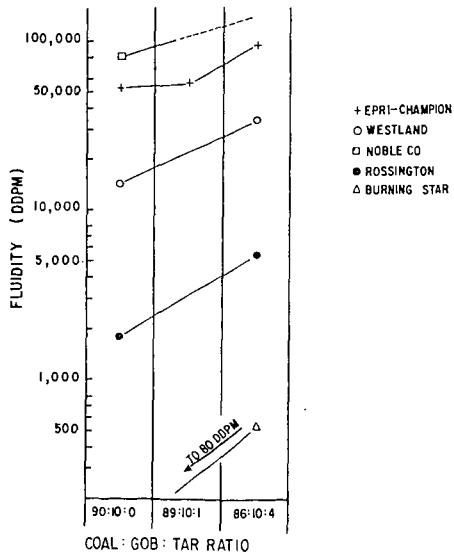


Figure 7  
Pressurized Swelling Index Apparatus

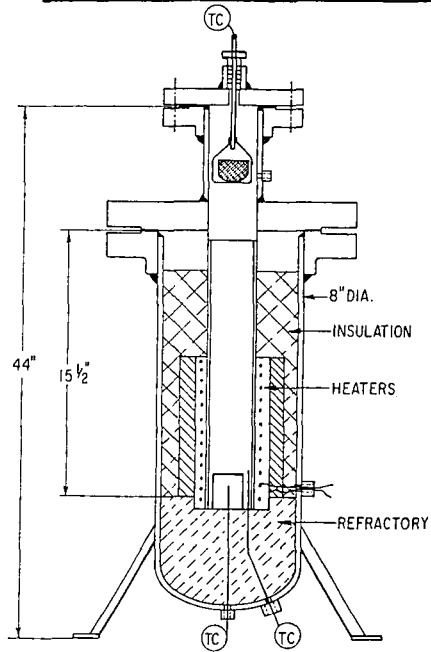


Figure 8  
EFFECT OF SAMPLE COMPOSITION ON THE SWELLING OF VARIOUS COALS

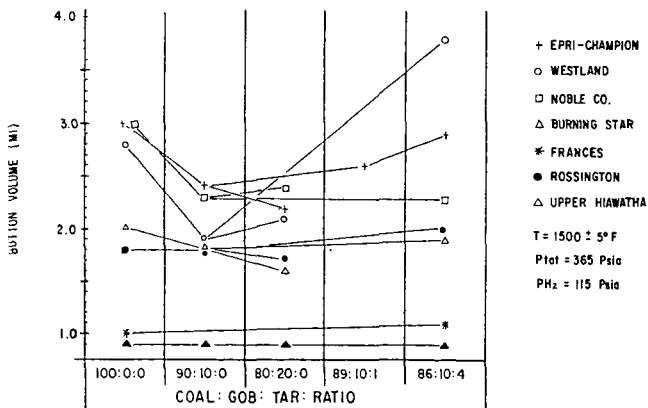


Figure 9  
SWELLING PROPERTIES OF PITTSBURGH NO. 8 & ILLINOIS NO. 6 COALS

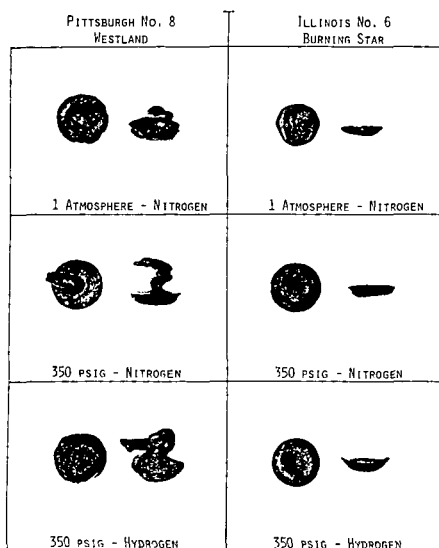


Figure 10  
Pressurized Coker

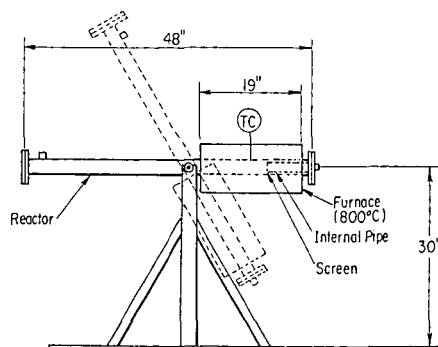


Figure 11  
EFFECT OF SAMPLE COMPOSITION, TOTAL PRESSURE AND  
HYDROGEN PRESSURE ON THE COKE STRENGTH

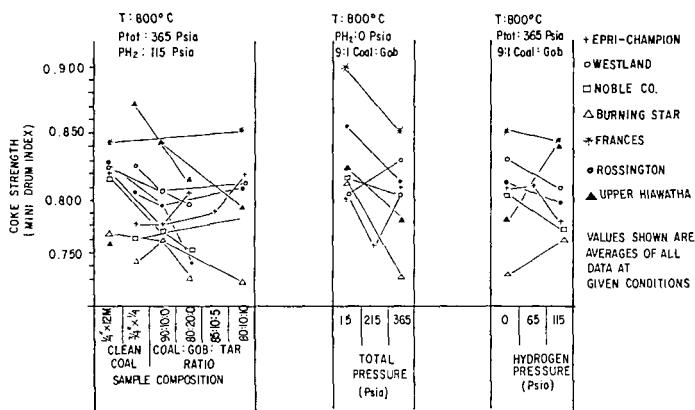


Figure 13  
EFFECT OF TOTAL PRESSURE, HYDROGEN PRESSURE AND  
SAMPLE COMPOSITION ON  $H_2$ , CO AND  $CH_4$  CONTENT IN  
PRODUCT GAS

